Microstructure Variation in Pilot Oven Coke

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INTRODUCTION

Techniques of microstructure analysis have been employed in defining coke types and quality for some time.[1,*2,3] However, there has been little effort expended to determine the microstructure variations between samples taken from various locations in large coke masses or the number of fingers that should be analyzed to adequately characterize coke structure. Coke microstructure data have been useful in explaining variations in physical properties,[2] reactivity,[3] and blast furnace behavior[4] of various coke types. Therefore, it is important that the limitations and reproducibility of microstructure analysis be determined if coke microstructure analyses are to be used to quantitatively characterize coke quality and behavior. Consequently, this investigation was initiated to determine the location and number of coke fingers needed to characterize the microstructure of coke produced in a 750-pound pilot coke oven and to evaluate our present coke sampling procedures.

As part of The Youngstown Sheet and Tube Company's program of coke evaluation, two duplicate charges of 100 per cent high volatile A bituminous coal were carbonized for this investigation. Samples were collected from each charge according to a statistically designed sampling plan. The coke finger samples were mounted and polished and their microstructure determined utilizing a six spindle integrating stage. The resultant microstructure data were then statistically evaluated.

PREPARATION OF COAL FOR CARBONIZATION

In order to provide coal for two duplicate charges, a bulk sample (1500 lbs.) of high volatile A bituminous coal was collected. This bulk sample (1" x 0" fraction) was pulverized to approximately 82 per cent minus 1/8" (Table I) in a Pennsylvania Type C reversible hammer-mill-impactor. All of the pulverized coal was then blended in a Model GD, Patterson-Kelly, 30 cubic foot capacity twin shell blender for two hours. After homogenization of the coal, samples were removed for screen and chemical analyses. Prior to withdrawing the coal for carbonization, the bulk density and moisture were adjusted and the coal was blended for an additional hour.

CARBONIZATION

Duplicate carbonization tests were made in a Bethlehem type pilot coke oven having a coking chamber 36 inches long, 18 inches wide, and 36 inches high. To insure duplication of these tests, the oven was stabilized for 24 hours before each charge. At charging time, a 750-pound sample of coal was withdrawn from the blender and placed in the oven. Pertinent carbonization data for these oven tests are given in Table I.

SAMPLING PROCEDURE AND EXPERIMENTAL DESIGN OF SAMPLING PLAN

A sampling plan was selected in order to provide data for an adequate appraisal of the microstructure of the resultant coke, as well as providing data for a

* See References.

comprehensive statistical evaluation. Although twelve oriented samples and one quench car "grab" sample were collected from each charge, only six oriented samples and the quench car sample were examined for each charge. This procedure was followed since it was believed that one sample from a particular location on the fixed wall would be the mirror image of the one immediately across from it on the movable wall or vice versa. The areas from which the samples were taken are shown on Figure 1. The remaining twelve samples were retained as reserve samples for additional studies. The coke side and push side samples were pulled from the charge with steel tongs after the "horseshoe" had been raised. The center samples were collected after half of the charge had been pushed from the oven. Each coke finger was immediately water quenched and labeled. The quench car sample was collected after the entire charge had been pushed and quenched.

Depending on viewpoint, the experimental design of the sampling plan can be considered either as (1) a full 3 x 2 x 2 factorial with replicates, or as (2) a fractional 3 x 2 x 2 x 2 factorial with replicates. The reason for the above difference depends on whether one considers the location of "mirror image" coke fingers as a replication or as a factor. This location refers to whether the coke finger sample was taken from the "fixed wall" or "movable wall" side of the oven. The above consideration was only of secondary importance in the initial analysis since samples had been taken for the full 3 x 2 x 2 x 2 factorial. However, only half of the original samples were microscopically analyzed for the initial analysis but the other half can be analyzed at a later date if it should prove statistically necessary to do so. Thus, the initial analysis distinguishes the confounded effect of charge and oven side rather than the independent effect of each. The factors referred to in the 3 x 2 x 2 x 2 factorial are (1) oven length location at three levels, (2) oven height location at two levels, (3) oven charge at 2 levels, and (4) oven side location at two levels. The experiment was designed to satisfy two objectives:

1. To determine the inherent errors of the various measurement categories associated with a microscopic analysis of coke.

2. To determine whether sampling errors were attributable to oven location and to determine if the present sampling procedures are adequate.

With respect to the first objective, it was necessary that a replicate transect be made on each coke finger, such that an adequate estimate of the "within coke finger" variability could be obtained. Although only 12 coke finger samples were analyzed, a total of 24 transects were taken by making two independent transects per coke finger. Thus, the "within coke finger" variability of each of the categories is based on 12 degrees of freedom and can be considered as the pooled within variance of the six locations and the two oven charges.

With respect to the second objective, it was necessary that samples be taken at various locations within the oven and also from more than one oven charge, such that it would be possible to determine whether differences due to oven location were independent of the oven charge. Since two transects per coke finger were necessary to satisfy the first objective and two oven charges were also necessary, it was felt that only six oven locations could be incorporated into the plan because of the time involved in making the individual transects. For this reason, the six oven locations were chosen, such that they would reflect large rather than small location differences and also provide an adequate representation of the entire oven. The oven was divided into a cross section of three vertical and three horizontal plots for a total of nine plots, but only the top three and the bottom three plots were used in the plan for a total of six samples per charge. The samples were then taken at random from somewhere within each of these six locations for each of the two charges.

The data were analyzed by use of the analysis of variance technique, such that the various sources of variability could be distinguished and compared.

The .05 significance level was used throughout the analysis to determine whether observed differences were "real" or not. For the purpose of this report, a "real" difference indicates that the risk of making an incorrect decision based on this significance level would be five times in 100.

SAMPLE PREPARATION

A section one-half inch thick was cut from the center through the entire length of each finger. In all cases, the full length of each finger (approximately nine inches), from the cauliflower to the tar end was maintained. The width of the fingers varied from two to three inches. To facilitate ease of handling, mounting, and polishing, the 9-inch slabs were cut in half. These sections, each representative of 1/2 finger, were then placed in molds, impregnated with an epoxy resin, and polished for microstructure analyses.

ANALYTICAL PROCEDURES

A Leitz Ortholux microscope (Figure 2) equipped with a Leitz six spindle integrating stage, cross hair, and an ocular micrometer was employed in these microstructure analyses. All of the measurements were carried out at 160X magnification. Four transects covering the entire length, from the cauliflower to the tar end, were completed on a single surface of each finger. Each transect was oriented perpendicular to the cauliflower or tar end, depending upon which half was being examined. During the first transect, the pores which fell within five arbitrarily chosen size categories were recorded on five of the spindles. The sixth spindle was used to record the total cell wall area transversed during the transect. At the end of the initial transect, the specimen was returned to the original starting position. A similar transect was then completed over the same area, and the cell wall sizes were differentiated and the total space occupied by pores was recorded on the sixth spindle. This procedure was repeated twice on each finger. The arbitrarily chosen categories (Table II) were selected from those presented by Abramski and Mackowsky.[2] The average cell wall thickness and pore diameter calculations (Table III) are those proposed by Daub[2] and are based on the supposition that the true average for any of the categories approaches that of the mid-point of the particular category in question. Although the authors have not completed a statistical confirmation of this assumption, they do feel it is a reasonable estimate of the average. The density values are expressed in terms of the ratio of the volume per cent of pores to cell walls in the coke.

RESULTS AND DISCUSSION

In Table I, the carbonization data for both charges indicate that the coal charges were subjected to essentially identical coking conditions. The thermocouple readings (Tables IV, V) taken at charging and pushing time do exhibit some variation. However, these temperature differences are normal for the oven in question, and ovens of this size and type. The statistical evaluation presented herein has not included the specific effect of temperature variation. However, it would be possible in future work to determine statistically the effects of temperature variation and oven location on coke microstructure by an analysis of co-variance of these factors.

A high volatile A bituminous coal was selected for this study because previous studies have shown that this rank of coal would produce a coke possessing considerable variation in microstructure. The authors believe that the variation in the microstructure of this coke type would be greater than that obtained on coke from other coals used at our Company coke plants. In general, higher rank metallurgical coals produce cokes with larger percentages of cell walls and pores per unit area and it is reasonable to assume that the degree of reliability would increase with increasing rank of the coal. This observation has been confirmed in our laboratory and is based

on frequency measurements of cell walls and pores in cokes produced from various ranks of coal.

In Table VI, the variability limits at the 95 per cent confidence level for the cell wall and pore diameter categories are given. In addition, the limits are given for the average cell wall thickness, average pore diameter, and density measurements. The "within finger" variability consists of the inherent variability of the material, plus the analytical variability.

The within variability limits listed for the 95 per cent confidence level are the limits developed for comparison of individual fingers, each of which was transversed twice. The "within finger" variabilities of the various measurement categories were statistically compared, and it was found that:

1. The within variability of the three cell wall categories of 0.1 to 0.2 mm, 0.2 to 0.5 mm, and +0.5 mm did not differ significantly among themselves. Also the three pore diameter categories of 0.2 to 0.5 mm, 0.5 to 1.0 mm, and +1.0 mm did not differ significantly among themselves. It is also interesting to note that there was no significant difference between these cell walls and pore diameter categories.

2. The two cell wall categories of -0.05 mm and 0.05 to 0.10 mm did not differ significantly from each other nor from the two pore diameter categories of -0.1 mm and 0.1 to 0.2 mm which also did not differ significantly from each other.

3. In comparing the categories in 2 above, the -0.05 mm cell walls and both of the pore diameter categories were significantly different at the .05 significance level from all the categories in 1 above. However, the 0.05 mm to 0.10 mm cell walls were only significant at the .10 level from the categories in 1 above, which would not be considered significant in this analysis.

Even though the analytical variability was not separated out of the "within finger" variability, it is believed that it would be much smaller than the "within" limits. Variations due to the positioning of the transect on the coke fingers were noted, but it was not separated out in these statistical analyses.

The values listed under "total variability" are the limits of reproducibility that would be obtained if a completely random finger were analyzed without any knowledge of its original position in the oven or the charge from which it was sampled. These limits are much greater than those given for the "within finger" variability. Therefore, it is important that the microscopist realize that this variability exists when microstructure analyses are completed on a completely random, pilot oven finger. The degree of reproducibility is reduced markedly when such a finger is analyzed. It is also interesting to note that for the two cell wall categories of 0.05 mm to 0.10 mm and 0.1 mm to 0.2 mm, the sampling variability due to oven location and charge was not significantly different from the variability within a single coke finger. This degree of homogeneity was not found to exist in any of the other categories.

In Table VII, the mean value for the twelve fingers taken from both charges are compared to the mean value for various locations in the oven. The average pore diameter for the top fingers was significantly larger than that of the bottom fingers. The percentages of pores in the -0.1 mm, 0.1 to 0.2 mm, and 0.2 to 0.5 mm categories were found to be significantly smaller in the samples from the top of the oven as compared to the samples from the bottom (Figure 3). For the +1.0 mm pore diameter category, the samples from the top of the oven had a significantly larger percentage than those from the bottom. Although the 0.5 to 1.0 mm category had a larger percentage

for the top samples than those from the bottom, this difference was not sufficiently large to be termed significant. Although the height in the oven had significant and marked effects on the pore diameter measurements, the height in oven was found to have little or no significant effects on the cell wall measurements (Figure 3).

The low density values for the top fingers are directly related to the increase in the percentage of large pores and total area occupied by pores. A highly significant difference was found between the density of the top and bottom samples. The density is expressed in terms of the ratio of the volume per cent of pore space to that occupied by cell walls. The increase in pore diameter and larger pores may be related to a decrease in bulk density of the coal at the top of the oven. The loosely packed coal would permit the formation of frothy coke. It is less restricted in the plastic state, and gas evolution would proceed more freely in the upper portion of the charge than at the bottom. These data also show that a given coal may produce a coke with markedly different microstructures within the same coke mass.

The variations in the microstructure of the coke side, middle, and push side samples are shown in Table VII and illustrated graphically in Figures 4 and 5. The increase in average cell wall thickness toward the center from both ends of the oven may be the result of variations in bulk density. The coal in the center of the oven would have a higher bulk density, since it lies directly below the charging hole, whereas, the coal next to the doors would have a lower bulk density.[5,6] Note in Figure 4 that the distribution of the various cell wall sizes varies from the ends to the middle fingers. It was found that this difference was significant, however, for only two cell wall categories: the -0.05 mm and the 0.2 to 0.5 mm categories. In Figure 5 the percentage of plus 1.0 mm pores for the end samples was significantly higher than that of the middle fingers. Coincident with the increase in larger pores, is the decrease in density of the end fingers (Table VII). This decrease in density is also statistically affected by the height-length relationship. All of these data indicate that coal bulk density, oven temperature and other variables not considered here may affect the resultant microstructure.

In Table VIII the microstructure analyses of each charge are listed. The significance of these data are as follows:

1. The mean values for the microstructure analyses of the six fingers taken from each of the charges compared quite favorably and did not exhibit any significant statistical differences.

2. Apparently the carbonization conditions and the composition of the coal were similar, as a result reproducible data were obtained.

3. Even though there are microstructure variations, the mean values are remarkably similar.

In order to determine if our present sampling plan was adequate to characterize a 750-pound charge, routine "grab" samples were collected and analyzed. Our present sampling of coke for microstructure analysis is as follows:

1. The entire charge is pushed and quenched.

2. A single finger exhibiting a cauliflower and a tar end and four fractured surfaces is selected from the top of the quench car by the lab technician.

3. It is labeled and forwarded to the anthracology laboratory for analyses.

In Table VIII and Figures 6 and 7, microstructure analyses of the two "grab" samples are compared to the mean values for their respective charges. These data show that all the values except the -0.05 mm cell wall category for the second "grab" sample, fall

within the 95 per cent confidence limits. This is true only if two transects are completed on each "grab" sample. Although the "grab" sample from the second charge is statistically outside the variability band in the -0.05 mm category by 0.21 per cent this could legitimately be adjusted by taking two "grab" samples from the quench car and making two transects on each sample. The authors feel that this minor variation is insignificant in view of the magnitude of the measurements involved and the practical aspects of these data. It is interesting to note that an examination of the four sample locations (top middle, bottom middle, top push side, and bottom push side) indicated that a sample representative of the charge could be obtained at a position somewhere within the central third plot and between the push side and middle third plots of the oven. This, incidentally, was the approximate area from which the present "grab" sample was being taken. Consequently, the present method of selectively collecting coke samples for microstructure analysis should be continued. However, this present method should be checked periodically by making additional transects on samples taken from the six locations as defined in the experimental design.

In Table IX the microstructure of cokes produced from various ranks of coal and petroleum, as well as form coke, are compared to the microstructure of the high volatile coal used in this study. Although there are no differences in certain cell wall and pore categories, there are marked differences among other categories. There are significant differences in average cell wall thickness and average pore diameter among the coke types and in certain categories a relationship to coal rank is evident.

SUMMARY

Microstructure analyses were completed on 12 oriented coke fingers collected from duplicate pilot oven tests of a single high volatile A bituminous coal. A selected "grab" sample was also collected from each charge and its microstructure determined. Duplicate transects were made on all coke fingers. These data show that from a practical viewpoint an analysis of a single selective sample adequately characterized the microstructure of coke produced in the 750-pound oven. The criterion of adequateness was satisfied, from a statistical viewpoint, in all but one of the measurement categories on one of the oven charges. Two selected "grab" samples should completely satisfy the adequateness criterion. Even though variations were noted because of sample location in the oven, the single "grab" samples compare favorably with the mean values for the charges.

REFERENCES

1. Krajewski, J., "Microscopic Research on the Structure of Coke Produced from Various Polish Coals," Translated from Polish, Builetyn Instytutu Naukowo-Badawezego Przemslu Weglowego, Report No. 37, 1948, pp. 1-25.

2. Abramski, C., and M. Th. Mackowsky, "Methoden und Ergebnisse der Angewandten Koksmikroskopie, H. Freund, Hanbuch der Mikroskopie in der Technik, Bank II, Teil I, Umschau Verlag Frankfurt am Main, 1952; pp. 311-410.

3. Schapiro, N., and R. J. Gray, "Relation of Coke Structure to Reactivity," Blast Furnace and Steel Plant, Vol. 51, No. 4, April 1963, p. 273.

4. Chang, M. C., and G. H. Denton, "Quality Considerations for Future Blast Furnace Coke," Paper presented at the Ironmaking Conference of the Metallurgical Society of AIME, Buffalo, New York, April 1-3, 1963.

5. Brisse, A. H., and J. G. Price, "Explorations in Cokemaking Research with a Full Scale Coke Oven Model," Blast Furnace and Steel Plant, Vol. 47, No. 12, December, 1959, pp. 1285-1290.

6. Zubilin, I. G., "Investigation into the Density Distribution of the Coal Charge in an Experimental Large-Capacity Oven Chamber," Coke and Chemistry, USSR, No. 6, 1961, pp. 22-26.

TABLE I - CARBONIZATION DATA

	1ST CHARGE	2ND CHARGE
Net charge weight	665 lbs.	673 lbs.
Net coking time	18 hrs. 37 min.	18 hrs. 45 min.
Oven bulk density	47.50 lbs./Ft.^{3}	48.07 lbs./Ft.^{3}
Kopper's cone	42.0 lbs./Ft.^{3}	40.6 lbs./Ft.^{3}
Per cent moisture	5.4	5.5
Per cent pulverization	82.8% - 1/8 inch	82.8% - 1/8 inch
Controlling Flue temp.	2460°F	2460°F
ASTM stability factor	43.54	42.14
ASTM hardness factor	55.35	55.55

TABLE II - SAMPLE CALCULATIONS OF PERCENTAGES OF PORES AND CELL WALLS IN THE INDICATED CATEGORIES

CELL WALL SIZE CATEGORIES	SPINDLE VALUES (mm)	PER CENT
0 - 0.05 mm	13.81	15.45
0.05 - 0.1 mm	16.36	18.31
0.1 - 0.2 mm	32.54	36.41
0.2 - 0.5 mm	24.15	27.03
+ 0.5 mm	2.50	2.80
Total cell wall	89.36	100.00

PORE SIZE CATEGORIES	SPINDLE VALUES (mm)	PER CENT
0 - 0.1 mm	21.90	8.59
0.1 - 0.2 mm	18.51	7.26
0.2 - 0.5 mm	59.99	23.55
0.5 - 1.0 mm	49.93	19.59
+ 1.0 mm	104.50	41.01
Total pore	254.83	100.00

TABLE III - SAMPLE CALCULATIONS OF AVERAGE PORE DIAMETER AND CELL WALL THICKNESS

CELL WALL SIZE CATEGORIES	SPINDLE VALUES (mm)		MID POINTS (MP)		NUMBER OF CELL WALLS
0 - 0.05 mm	13.81	÷	0.025		552.40
0.05 - 0.1 mm	16.36	÷	0.075		218.13
0.1 - 0.2 mm	32.54	÷	0.15		216.93
0.2 - 0.5 mm	24.15	÷	0.35		69.00
+ 0.5 mm	2.50	÷	0.75		3.33
Total cell wall	89.36 mm			Total	1059.79

Average cell wall thickness = $\frac{89.36}{1059.79}$ = .084 mm

PORE SIZE CATEGORIES	SPINDLE VALUES (mm)		MID POINTS (MP)		NUMBER OF PORES
0 - 0.1 mm	21.90	÷	0.05		438.00
0.1 - 0.2 mm	18.51	÷	0.15		123.40
0.2 - 0.5 mm	59.99	÷	0.35		171.40
0.5 - 1.0 mm	49.93	÷	0.75		66.57
+ 1.0 mm	104.50	÷	1.50		69.67
Total pore	254.83 mm			Total	869.04

Average pore diameter = $\frac{254.83}{869.04}$ = .293 mm

TABLE IV - COMPARISON OF TEMPERATURE READINGS TAKEN AT CHARGING TIME

THERMOCOUPLE	1ST CHARGE FIXED WALL	2ND CHARGE FIXED WALL	1ST CHARGE MOVABLE WALL	2ND CHARGE MOVABLE WALL
1	1870°F	1850°F	1760°F	1760°F
2	1870	1860	1820	1800
3	1850	1840	1800	1840
4	1950	1960	1890	1900
5	1900	1890	1900	1880
6	out	out	1820	1860
7	1780	1700	1860	1860
8	1860	1840	1920	1910
9	1850	1880	1600	1610
Average	1866°F	1853°F	1819°F	1824°F

TABLE V - COMPARISON OF TEMPERATURE READINGS TAKEN AT PUSHING TIME

THERMOCOUPLE	1ST CHARGE FIXED WALL	2ND CHARGE FIXED WALL	1ST CHARGE MOVABLE WALL	2ND CHARGE MOVABLE WALL
1	1860°F	1800°F	1890°F	1840°F
2	2000	1900	2070	2060
3	2020	2020	1980	1990
4	1980	1970	2000	1970
5	2140	2110	2170	2150
6	out	out	2140	2100
7	1760	1770	1940	1890
8	1980	1840	2040	2020
9	1930	1870	1880	1800
Average	1959°F	1910°F	2012°F	1980°F

THERMOCOUPLES IN COKE MASS	1ST CHARGE	2ND CHARGE
#3 Fixed wall	1920°F	1900°F
#4 Movable wall	1920	1900
#5 Center	1890	1870

TABLE VI - "WITHIN FINGER" AND TOTAL VARIABILITY ERRORS AT THE 95 PER CENT CONFIDENCE LEVEL FOR ONE AND TWO TRANSECTS

CATEGORIES	"WITHIN FINGER" VARIABILITY		TOTAL VARIABILITY*	
	PER CENT		PER CENT	
CELL WALLS	ONE TRANSECT	TWO TRANSECTS	ONE TRANSECT	TWO TRANSECTS
- 0.05 mm	± 2.477	± 1.752	± 5.169	± 5.014
0.05 - 0.1 mm	± 3.716	± 2.628	± 3.569	± 3.022
0.1 - 0.2 mm	± 5.596	± 3.957	± 5.849	± 5.102
0.2 - 0.5 mm	± 5.553	± 3.926	± 8.275	± 7.773
+ 0.5 mm	± 5.900	± 4.172	± 6.327	± 5.562
PORES				
- 0.1 mm	± 1.782	± 1.260	± 4.933	± 4.849
0.1 - 0.2 mm	± 2.777	± 1.964	± 8.248	± 8.124
0.2 - 0.5 mm	± 5.127	± 3.626	±13.101	±12.197
0.5 - 1.0 mm	± 6.119	± 4.327	± 8.804	± 8.230
+ 1.0 mm	± 5.288	± 3.739	±22.487	±22.324
Average Cell Wall Thickness mm	.00919	.00650	.01554	.01482
Average Pore Diameter mm	.02534	.01792	.08957	.08863
Density**	.08669	.0613	.1731	.1673

* Total variability error: expect error if a random sample is analyzed without any knowledge of its original position in the oven.

** Ratio of volume per cent pore area to volume per cent cell wall area in coke.

TABLE VII - COMPARISON OF MEAN VALUES BY SAMPLE LOCATION AND CATEGORIES TO THE GRAND MEAN FOR BOTH CHARGES

CATEGORY	GRAND MEAN FOR BOTH CHARGES	TOP SAMPLES	BOTTOM SAMPLES	COKE SIDE SAMPLES	MIDDLE SAMPLES	PUSH SIDE SAMPLES
CELL WALLS						
- 0.05 mm	14.96	14.57	15.34	14.16	13.71	17.00
0.05 - 0.1 mm	18.62	18.79	18.45	18.73	18.15	18.98
0.1 - 0.2 mm	33.43	33.62	33.25	33.86	34.02	32.42
0.2 - 0.5 mm	28.24	28.15	28.33	29.89	29.46	25.37
+ 0.5 mm	4.75	4.87	4.63	3.36	4.66	6.23
Per Cent	100.00	100.00	100.00	100.00	100.00	100.00
Average Cell Wall Thickness mm	0.087	0.088	0.086	0.089	0.091	0.082
PORES						
- 0.1 mm	10.68	8.63	12.72	10.80	11.30	9.92
0.1 - 0.2 mm	13.66	10.08	17.25	12.94	14.11	13.94
0.2 - 0.5 mm	31.15	25.84	36.47	29.51	32.71	31.24
0.5 - 1.0 mm	24.43	25.51	23.35	24.77	24.82	23.71
+ 1.0 mm	20.08	29.94	10.21	21.98	17.06	21.19
Per Cent	100.00	100.00	100.00	100.00	100.00	100.00
Average Pore Diameter mm	0.236	0.275	0.196	0.238	0.223	0.247
Density (Vol. % Cell Walls / Vol. % Pores)	0.466	0.398	0.534	0.471	0.486	0.441

TABLE VIII - COMPARISON OF EACH GRAB SAMPLE TO THE MEAN VALUES FOR THE RESPECTIVE CHARGES

CATEGORY	MEAN 1ST CHARGE	LIMITS	1ST CHG. GRAB SAMPLE	MEAN 2ND CHARGE	LIMITS	2ND CHG. GRAB SAMPLE
CELL WALLS						
- 0.05 mm	14.08	15.83 - 12.33	12.65	15.82	17.57 - 14.07	13.86*
0.05 - 0.1 mm	18.97	21.60 - 16.34	18.76	18.21	20.84 - 15.58	16.89
0.1 - 0.2 mm	34.18	38.14 - 30.22	36.21	32.67	36.63 - 28.71	35.86
0.2 - 0.5 mm	29.09	33.02 - 25.16	29.44	27.48	31.41 - 23.55	28.81
+ 0.5 mm	3.68	7.85 - 0	2.94	5.82	9.99 - 1.65	4.58
Per Cent	100.00		100.00	100.00		100.00
PORES						
- 0.1 mm	10.58	11.84 - 9.32	11.14	10.74	12.00 - 9.48	9.97
0.1 - 0.2 mm	13.61	15.57 - 11.65	12.56	13.68	15.64 - 11.72	14.87
0.2 - 0.5 mm	31.92	35.55 - 28.29	29.68	30.42	34.05 - 26.79	30.26
0.5 - 1.0 mm	24.41	28.74 - 20.08	24.29	24.46	28.79 - 20.13	24.35
+ 1.0 mm	19.48	23.22 - 15.74	22.33	20.70	24.44 - 16.92	20.55
Per Cent	100.00		100.00	100.00		100.00
Average Cell Wall Thickness mm	.089	.0955 - .0825	.092	.085	.0915 - .0785	.090
Average Pore Diameter mm	.233	.2509 - .2160	.228	.238	.2559 - .2201	.232
Density ($\frac{\text{Vol. \% Cell Walls}}{\text{Vol. \% Pores}}$)	.473	.534 - .412	.418	.459	.520 - .398	.459

* Only value which does not fall within the variability limits.

TABLE IX - VARIATION IN MICROSTRUCTURE OF INDICATED COKE TYPES.

CATEGORY	High Vol.	High-Med. Vol.	Low-Med. Vol.	Low Vol.	Petroleum Coke	Form Coke
CELL WALLS						
- 0.05 mm	13.25	16.09	18.84	29.49	0.19	15.45
0.05 - 0.1 mm	17.83	21.35	20.68	23.20	0.12	25.38
0.1 - 0.2 mm	36.04	38.71	34.33	27.89	1.03	40.50
0.2 - 0.5 mm	29.12	20.49	18.79	13.67	6.52	16.45
+ 0.5 mm	3.76	3.36	7.36	5.75	92.14	2.22
Per Cent	100.00	100.00	100.00	100.00	100.00	100.00
Average Cell Wall Thickness mm	0.091	0.080	0.076	0.058	0.635	0.078
PORES						
- 0.1 mm	10.55	17.23	15.62	23.62	1.98	23.76
0.1 - 0.2 mm	13.72	16.68	21.88	24.63	3.72	29.16
0.2 - 0.5 mm	29.97	37.66	40.09	37.20	15.03	34.91
0.5 - 1.0 mm	24.32	19.86	17.63	10.66	17.05	12.17
+ 1.0 mm	21.44	8.57	4.78	3.89	62.22	-
Per Cent	100.00	100.00	100.00	100.00	100.00	100.00
Average Pore Diameter mm	0.230	0.168	0.167	0.132	0.583	0.127
Density $\left(\frac{\text{Vol. \% Cell Walls}}{\text{Vol. \% Pores}}\right)$	0.439	0.569	0.589	0.534	1.59	0.747

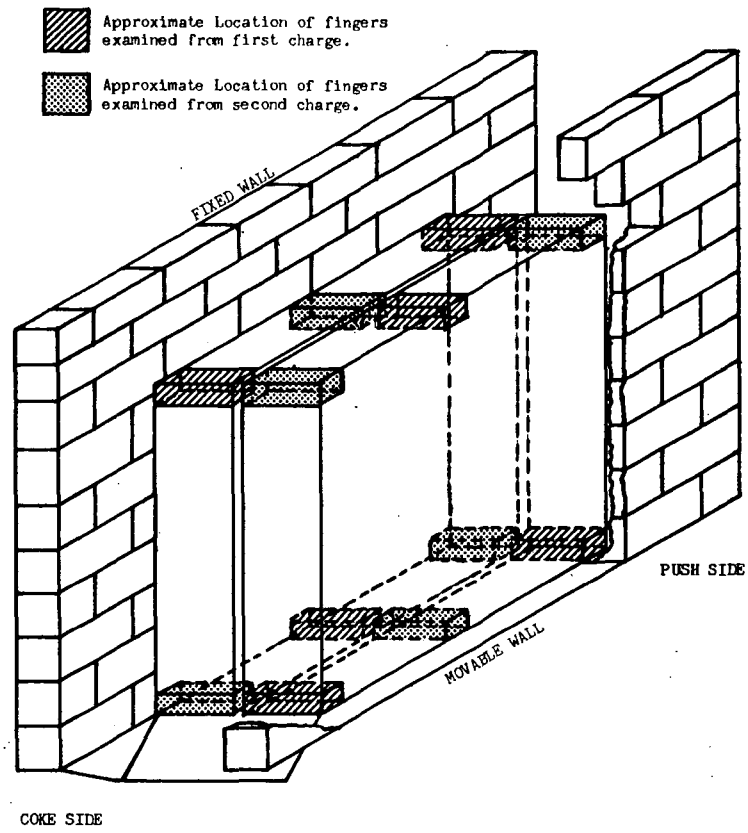


FIGURE 1 - SCHEMATIC OF 750 POUND OVEN SHOWING APPROXIMATE LOCATION OF COKE FINGERS EXAMINED FROM DUPLICATE CHARGES.

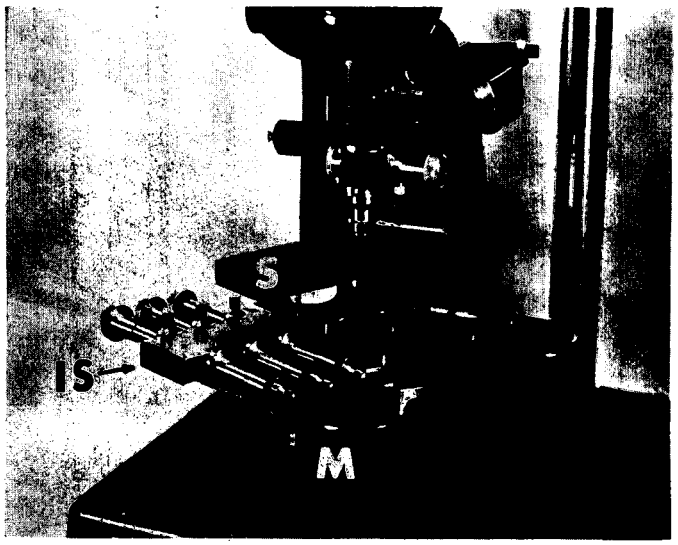


FIGURE 2 - PHOTOGRAPH OF MICROSCOPE AND INTEGRATING STAGE EMPLOYED IN MICROSTRUCTURE ANALYSES (1/4 X). INTEGRATING STAGE (IS), SPECIMEN (S), AND MICROSCOPE STAND (M).

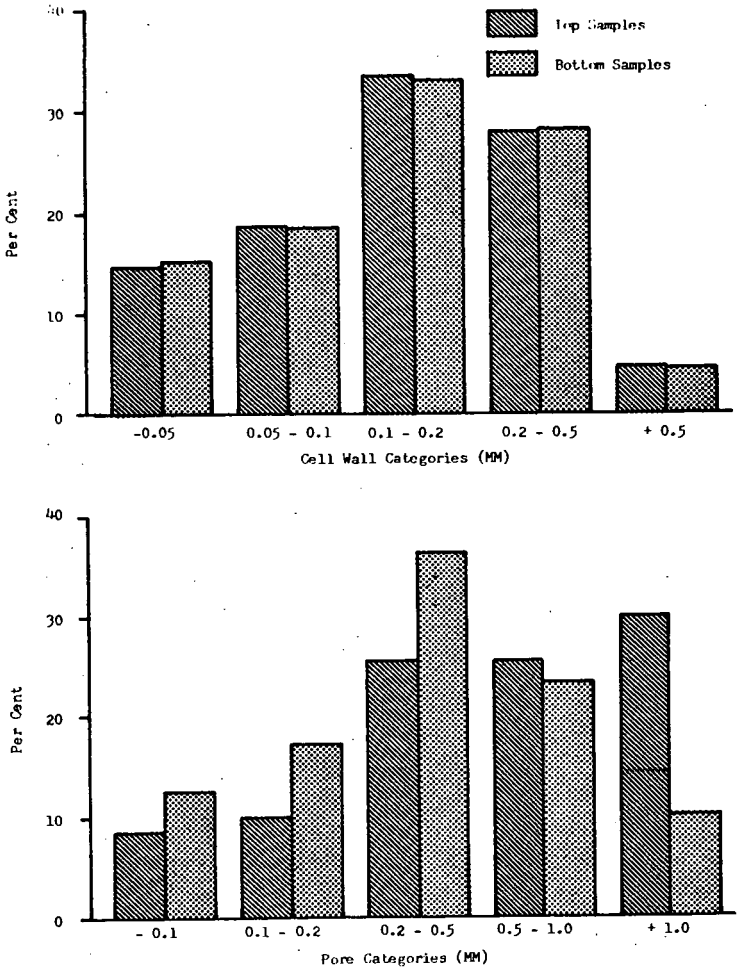


FIGURE 3 - COMPARISON OF FINGERS TAKEN FROM TOP OF THE OVEN TO THOSE TAKEN FROM BOTTOM OF OVEN FOR BOTH CHARGES

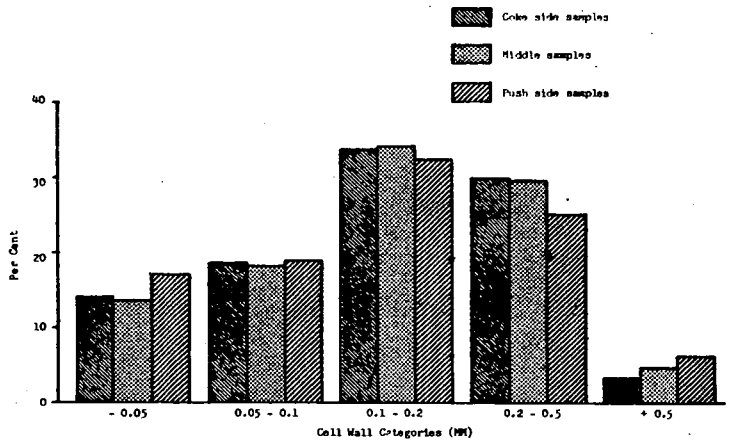


FIGURE 4 - COMPARISON OF PER CENT CELL WALLS IN THE INDICATED CATEGORIES FOR THE COKE SIDE, MIDDLE, AND PUSH SIDE SAMPLES.

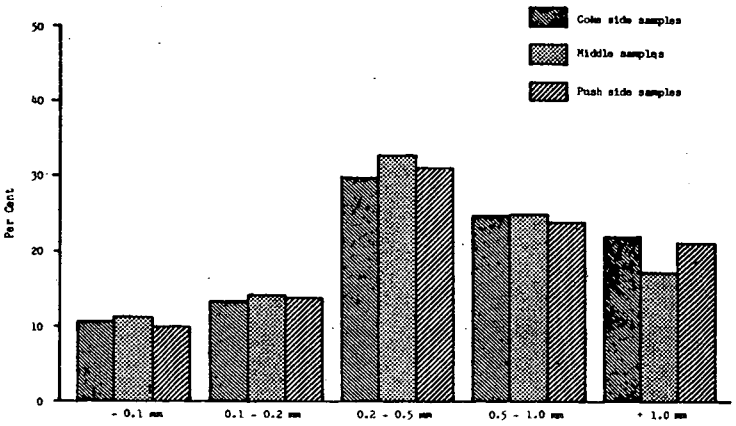


FIGURE 5 - COMPARISON OF PER CENT PORES IN THE INDICATED CATEGORIES FOR THE COKE SIDE, MIDDLE, AND PUSH SIDE SAMPLES.

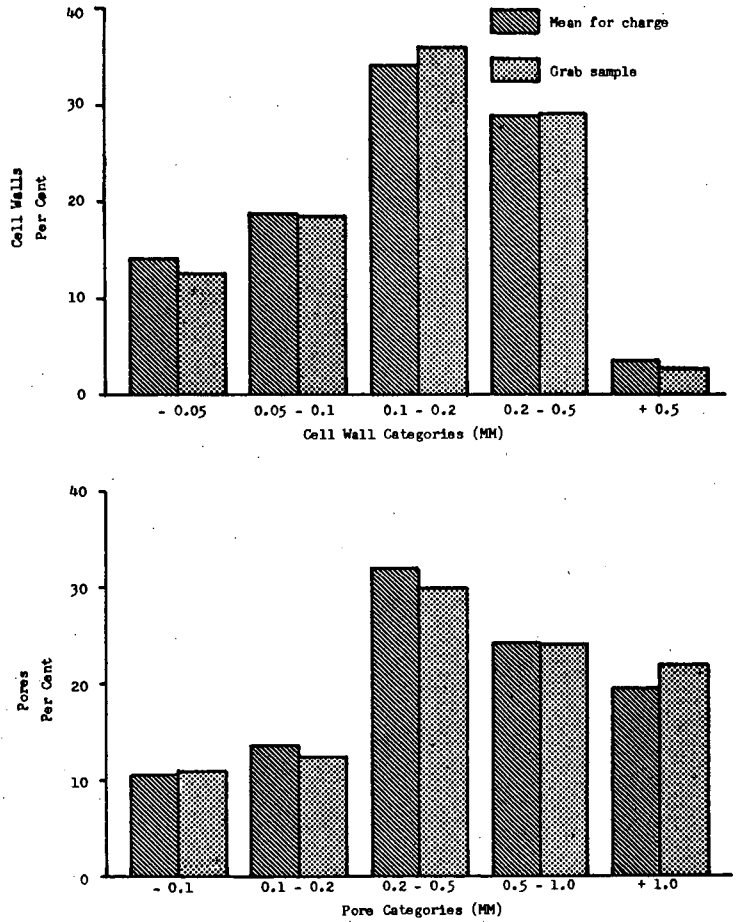
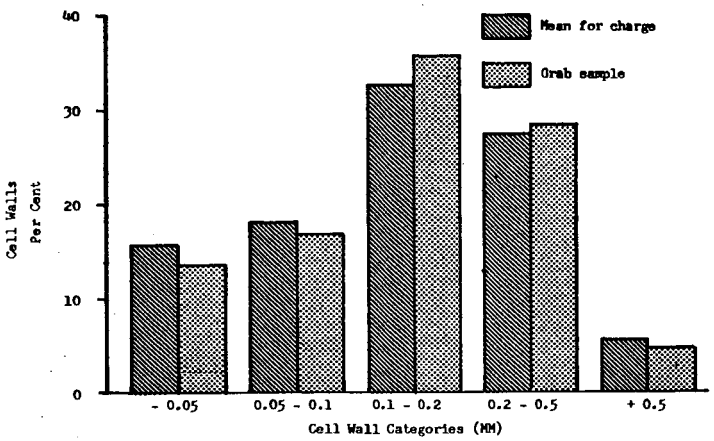


FIGURE 6 - COMPARISON OF MEAN VALUES FOR 1ST CHARGE TO THE MEAN VALUES FOR THE SINGLE GRAB SAMPLE TAKEN FROM THIS CHARGE.



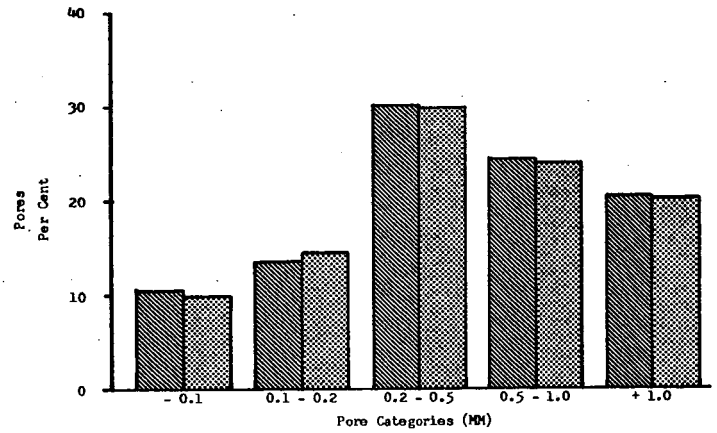


FIGURE 7 - COMPARISON OF MEAN VALUES FOR 2ND CHARGE TO THE MEAN VALUES FOR THE SINGLE GRAB SAMPLE TAKEN FROM THIS CHARGE.